PROGRESS REPORT
FRACTURE OF HARDENED CEMENT PASTE

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by

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Joint Highway Research Project

PURDUE UNIVERSITY
LAFAYETTE INDIANA

Progress Report

FRACTURE OF HARDENED CEMENT PASTE

To: Dr. G. A. Leonards, Director
Joint Highway Research Project

June 20, 1967

From: H. L. Michael, Associate Director
Joint Highway Research Project

File: 5-14-4
Project: C-36-61D

The attached progress report entitled "Fracture of Hardened Cement Paste" concerns the initial portion of the investigation of fracture phenomena in hardened portland cement paste. The investigation is being conducted by Mr. S. J. Hanna, Research Assistant, Joint Highway Research Project, under the direction of Dr. W. L. Dolch, Research Engineer, Joint Highway Research Project.

The initial portion of the investigation was concerned with the development of procedures and techniques both for making and for testing specimens. The examination of fractured surfaces has provided insight into the origin and propagation of critical cracks. A comparison of some initial test results with existing brittle fracture theory indicate a general agreement.

Respectfully submitted,

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By

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Purdue University
June 20, 1967

INTRODUCTION

Portland cement concrete is widely used as a structural building material in a variety of applications. In the area of highway transportation portland cement concrete is used as a structural element in the highway and in its attendant structures. If the concrete fails by fracture, there is a resulting loss in load-carrying ability, and in smoothness. Increased maintenance and repair costs result from such failures. From both an economic and safety standpoint, it is important to have a better understanding of the failure mechanisms of concrete. If these failure mechanisms are well-understood, steps can be taken in the design of the concrete mixture and in the structural design to reduce the probability of failure and the resultant costs of repair or replacement.

Much interest has been generated in recent years on the fracture mechanics of concrete and other ceramic materials. Investigations have been conducted to analyze the behavior of concrete under various stress conditions. The Griffith theory of brittle fracture (1) and the Orowan modification of it (2) have been used to explain the process of fracture in glasses and metals. Kaplan (3) has applied the Griffith theory, as modified by Irwin (4) to concrete. Concrete is a mixture of cement paste and aggregates, a very complex and inhomogeneous system. Cement paste itself is inhomogeneous owing to the presence of unhydrated cement particles, capillary pores, gel pores, and microcracks formed by thermal and shrinkage stresses. Such microcracks occur even prior to external loading.

In comparison with concrete the cement paste can be considered a relatively homogeneous material. The water-cement ratio is the primary factor controlling the strength of the paste through its controlling effect on the porosity of the hardened paste. The lower the water-cement ratio, the lower the porosity and the higher the strength. Other variables, such as aggregate properties, along with the water-cement ratio affect the strength of portland cement concrete. With a number of interrelated variables present, the effect of a single one is masked by the action of the others. Any investigation of the fracture phenomena in concrete is thus extremely complex. The complexity of the system is reduced when only the hardened cement paste is considered.

FRACTURE OF CEMENT PASTE

The objective of this investigation is to examine the tensile fracture phenomena in hardened portland cement paste; including crack origin, growth and inhibition. In the initial phase of the investigation the water-cement ratio and specimen size were variables. The results obtained will be interpreted by means of the modified Griffith and strain-energy-release rate theories to test their applicability and to derive more acceptable models for the fracture of this material.

Experimental Considerations

An examination of the fractured surface of a brittle material can aid in the determination of the source of the fatal crack and the direction of crack propagation. Since hardened cement paste specimens tend to shatter when tested in compression, it is frequently impossible to examine the fractured surfaces. Specimens tested in direct tension, such as the Briquet test, are easily subjected to eccentric loadings, which make accurate determinations of the stress at failure difficult. Specimens tested in flexure have a stress gradient across their cross section, varying from tensile on one side to compression on the other. For these reasons and other considerations it was deemed advisable to use a test which would not embody these objections.

The diametral-compression test (splitting tensile or Brazilian test) (5) has recently been used to evaluate tensile strength of concrete cylinders. This test overcomes some of the objections to the other types of tensile tests in that a relatively uniform stress state is developed over a major portion of the cross section.

A specimen shape developed by Durelli, Morse, and Parks (6) is designed to fail in tension in the central bar (see Figure 1). The specimen is theta-shaped and is loaded in the plane perpendicular to the central web. A specimen of this shape, when loaded, experiences only tensile forces in the central web, with no stress gradient across that section. The orientation of the specimen is easily controlled to prevent eccentric loading. The theta specimen also has the advantage of allowing a small section to be tested which is of value in determining the effect of specimen size on the fracture phenomena.

Analysis of the stresses, in both the diametral-compression specimen and the theta specimen, is based on elastic theory. Both types of tensile tests were used in this investigation to evaluate the "tensile strength" of hardened portland cement paste.

Small specimen sizes were selected to minimize problems of handling the specimens and to minimize the effects of shrinkage and thermal changes in the paste specimens. Large cement paste specimens are also difficult to manufacture with uniform densities (Antrim (7) and Mullen (8)). Cylinders with a diameter of one inch or less and lengths varying from 0.1 inch to over 2 inches were tested in diametral-compression. Theta specimens with a web thickness of 0.10 inches and depths varying from approximately one tenth inch to three tenths of an inch were tested. Preliminary results indicated that specimens of these sizes and shapes can be made and tested satisfactorily.

The small size of the paste specimens makes them sensitive to oils and greases used to prevent bonding of the cement paste to mold surfaces. Absorption of these materials into the paste affects the surface tension in the paste and hence the strength. With this in mind, materials which did not require a lubricant to break the bond were considered for the molds. Teflon and some other plastics are chemically inert to cement paste. It is also relatively easy to form smooth mold faces with these materials. Teflon was selected as a mold material since it is chemically inert, has a high coefficient of thermal expansion, and is easily machined. For the cylinder molds hollow teflon cylinders and plastic test tubes were used to provide cylindrical specimens that would not have mold joint marks on their surfaces which could affect the failure of the specimen. The temperature susceptibility of these mold materials made it possible to remove the specimens from the molds by simply heating the mold surfaces slightly thereby causing them to expand away from the specimen.

One possible source of flaws which initiate failure are the capillary pores in the hardened cement paste. To provide a range of capillarity, specimens were made with several different water-cement ratios. These ranged from a dense mix with a water-cement ratio of 0.3 to a less dense mix with a water-cement ratio of 0.6. Water-cement ratio was considered as a variable for the theta specimens as well as the cylinders, which were tested in both direct compression and diametral-compression.

In his original work, Griffith noted that as the glass was drawn to very thin fibers the observed strength approached that of the calculated strength based on intermolecular forces (1). The inference being that the smaller the cross section the lower the probability of a flaw existing, and being critically oriented. With this in mind, specimen size was considered as a variable. Cylinders of various diameters and lengths were tested in diametral-compression. The thickness of the theta specimens were also varied, primarily due to the polishing operation used to eliminate flaws on the surfaces.

The tensile strength of small specimens is greatly influenced by entrapped foreign substances. It has been shown by experimental stress analysis that stress concentrations of three or four (or more) times the average applied stress can occur when holes are present. Care was taken in handling the cement and water to prevent contamination with particles of sand and other particles. Any entrapped particle or void creates a stress concentration in the specimen when it is stressed. This was immediately obvious when theta specimens cast from paste mixed in a mechanical mixer were tested. Every specimen tested failed at entrapped air voids, or at obvious surface defects. To obtain a better understanding of the behavior of cement paste under tensile stress and to cause failure to occur at predetermined points (by insertion of artificial flaws of a known size and orientation) a method of mixing the paste was developed to prevent entrappment of air.

The mixing action of the mixer paddle whipped a relatively large amount of air into the fresh paste. An attempt was made to remove the air by placing the fresh paste under a vacuum thereby reducing the pressure and drawing the air out of the paste. The disadvantage of this procedure was that the fresh paste foamed and bubbled when the vacuum was applied. This method was also undesirable since water was also drawn from the specimen changing the water-cement ratio. This made control of the water-cement ratio practically impossible.

A method of mixing the cement paste under partial vacuum using deaired water and cement was developed and proven practical for mixes of the size required to cast three theta specimens and six cylinders. The entrapped air was practically eliminated by use of this mixing procedure.

Extreme care was still required when placing the fresh paste in the molds to prevent entrapment of air.

The paste specimens were placed in lime water for curing upon removal from the molds. The specimens remained in the lime water until immediately prior to placement of the specimen in the testing machine. Care was taken at all times to prevent the specimens from drying and thereby inducing shrinkage stresses.

All testing was performed with a constant rate of vertical head travel of 0.05 inches per minute in the initial portion of this investigation. This rate was carefully checked each time a series of tests were performed since rate of strain (or rate of loading) does have an influence on the magnitude of the load at failure. A hydraulic testing machine with two load cells (range 0 to 500 pounds and range 0 - 5000 pounds) was used to test the theta and diametral-compression specimens. A brushpen recorder and an X-Y recorder were used to record accurately the loads and strains. The use of the X-Y recorder made possible direct plots of the load versus vertical deflection while the test was in progress. With the large number of tests performed these records saved much time in data evaluation and a permanent record of each test was available.

After a specimen was tested in tension, the fractured specimen was carefully stored in a container to keep it undamaged and available for microscopic examination. A binocular microscope with a range in power up to 120 was used to view the fractured surfaces. These surfaces were carefully checked and any obvious flaws (such as air voids) were noted as to size and position on the fractured face. This information was recorded along with other pertinent information on a data sheet. The extent and location of any change in surface texture on the fractured face was also noted. Changes in surface texture provide information about the source and direction of propagation of the fatal crack.

To provide a better insight into the source of critical cracks specimens were manufactured with artificial flaws. Initial tests involved the use of thin teflon disks placed in the central portion of the paste cylinders to be tested in diametral-compression. Holes were also drilled through the central axis of some cylinders. If an artificial flaw much larger than any inherent flaw is present and oriented in the failure plane of a specimen then the effect of size of the specimen would be diminished or eliminated. Artificial defects were also introduced just at the ends of the cylinders in an effort to originate a critical crack at that point. Preliminary tests indicated that the fatal crack did originate at points where artificial flaws were placed.

Specimen Preparation

In the first part of the investigation the cement paste was mixed using an electric mixer. Since this method of mixing tended to produce a cement paste with a significant amount of entrapped air, a method of mixing the materials under a partial vacuum was used to reduce the entrapment of air in the paste. Care was taken to produce mixes of uniform consistency.

Materials

The materials used in this investigation were tap water, deionized water and Type I portland cement. The Type I portland cement was from a single clinker batch manufactured in central Indiana (laboratory designation 317). Table 1 shows the composition of the cement. It was assumed that the characteristics of the cement did not vary significantly since it was from a single clinker batch.

TABLE 1

PHYSICAL AND CHEMICAL PROPERTIES OF CEMENT 317

Physical Properties

Fineness, No. 325 Sieve	95.9 percent
Specific Surface, Blaine	3380 sq. cm. per gm.
Initial set	3 hr. 15 min.
Final set	5 hr. 05 min.
Air Entrained (ASTM Designation: C185-59)	8.5 percent

Chemical Analysis

Compound	Percentage present
Silicon Dioxide, SiO_2	21.76
Aluminum oxide, Al_2O_3	5.41
Ferric oxide, Fe_2O_3	1.97
Calcium oxide, CaO	65.30
Magnesium oxide, MgO	1.11
Sulphur trioxide, SO_3	2.43
Loss on ignition	1.78

Calculated Compound Composition

Compound	Percentage present
Tricalcium Silicate, C_3S	51.20
Dicalcium Silicate, C_2S	23.83
Tricalcium Aluminate, C_3A	11.00
Tetracalcium Aluminoferrite, C_4AF	5.99
Calcium Sulphate, $CaSO_4$	4.13

Mixing

For mixes produced under atmospheric pressure a Hobart mixer (Model N-50) was used. The mixer, paddle, and mixing bowl conformed to the requirements of ASTM Designation: C 305-64T, Tentative Method for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency.

The cement was carefully weighed and placed in the mixing bowl. The water was measured on a volumetric basis using a graduated cylinder. The water was city water which had been allowed to run from the tap for several minutes to stabilize the temperature. The water was added to the cement over a 10 second period with the mixer operating at the slow speed. Mixing continued at the slow speed for one minute. The speed was then increased to the medium speed for 30 seconds. The mixing bowl was removed at the end of that time and the paste mixed by hand for 30 seconds with a large mixing spoon. Care was taken to scrape any material from the sides and blend it into the mass. The bowl was replaced and mixing continued at the medium speed for another 30 seconds. At the end of this time the mixing bowl was covered and allowed to stand undisturbed for one minute and 30 seconds. The mixing was then continued at the medium speed for an additional minute. A total of five minutes elapsed between the addition of water and the completion of mixing.

For mixes produced under a partial vacuum, a plexiglass cylinder containing balls and rods for agitation of the paste was used as the mixer. The evacuated cylinder containing the cement and water was rotated on a roller mill to provide for the mixing action as the balls and rods agitated the cement paste. The plexiglass cylinder was approximately 12 inches high with a 6 inch diameter. The cylinder was sealed on one end and fitted with a screw-on-cap at the opposite end. The cap and the cylinder were threaded for a close fit and an O-ring seal was provided.

The cement was carefully weighed and placed in the mixing cylinder along with 12 flint balls (approximately one inch in diameter) six metal rods and three teflon rods. The end cap was then placed on the cylinder. Vacuum grease was placed on the O-ring and the upper portions of the threads to insure an air tight seal. A copper seat was provided in the permanently sealed end for the insertion of a stopcock which was held in place by a rubber stopper. The cylinder was then evacuated for fifteen minutes by means of a water operated aspirator. The cylinder was kept in an upright position for the first half of the period and then rotated several times to fluff the cement and placed on its side for the remainder of the fifteen minute period. At the end of this time the stopcock was closed, the aspirator hose detached and the vessel containing the water attached by means of a two inch section of rubber vacuum hose.

The water was deionized and had been deaired for fifteen minutes by boiling on an aspirator. The vessel containing the water was a calibrated filter funnel with a ground glass stopcock and a ground glass stopper. The water was placed in the vessel taking care not to trap air in the water. Water was introduced into the tubing between the cylinder and the filtering funnel, the funnel attached, and the stopcock of the funnel opened to allow any air which was trapped to escape to the top of the funnel. The stopcock on the cylinder was then opened and the water allowed to be pulled into the mixing cylinder until the proper amount had entered, at which time the cylinder stopcock was closed and the connecting hose detached. The calibrated filtering funnel allowed for an accurate determination of the water entering the cylinder.

The mixing cylinder was immediately placed on a Syntron-Jogger (Model PJ-15) to provide vibration and rotated by hand. This procedure provided the initial blending of the cement and water. The cylinder was placed on a roller mill, which rotated the cylinder at approximately 80 rpm, after three minutes of combination vibration and hand turning. After five minutes rotation, the cylinder was removed and hand turned and vibrated for two minutes before returning to the roller mill for an additional five minutes. The cylinder was then allowed to stand for two minutes before a final three minutes of rotation. The cylinder was then removed and the stopcock opened to admit atmospheric pressure.

Casting Specimens

The amount of cement (1000 g.) and the amount of water (dependent on the water-cement ratio) was selected so that sufficient cement paste would be available to cast three theta specimens and six cylinders. Immediately after mixing the mixing container was placed next to the molds and the paste carefully blended with a large mixing spoon to insure uniformity of the mix.

The theta molds were positioned on the vibrating table (Syntron-Jogger Model PJ-15) with the initial power setting at 50 on the scale for the filling period. One large mixing spoonful of paste was sufficient to fill each of the three theta molds. All molds were overfilled and a round nosed spatula was used to "rod" the paste. Care was taken to rod the paste at the paste-mold interfaced to force to the surface any entrapped air. The vibration was then increased to a power setting of 90 for one minute and the overfilled molds then set aside while the cylinder molds were filled.

The six cylinder molds were placed on the vibrating table with the power setting at 50 during the filling operation. The cylinder molds were filled in three layers with each layer rodded 25 times. The vibrating table power setting was increased to 90 and the six cylinders vibrated for three minutes. Rubber stoppers were then placed in the tops of the molds taking care not to trap air and the molds were inverted to vibrate for three minutes with the power setting at 50. The molds were allowed to stand for 20 minutes inverted. During the 20 minute period the theta specimens were again placed on the vibrating table with the power setting at 50. The theta specimens were struck off with a straight edge to provide a plane top surface. A final brief vibration with the setting at 90 provided the final leveling. Each theta specimen was placed in the fog room immediately after the final leveling with an inverted plastic container placed over them to allow air circulation in an atmosphere of 100 percent relative humidity and to prevent free moisture from dropping onto the specimen face.

At the end of the 20 minute standing period the cylinder molds were reinverted and vibrated for two minutes with the setting at 50. The molds were then allowed to stand for 8 minutes undisturbed after which they were again vibrated for two minutes in the inverted position. Another 8 minute standing period was provided before a final vibration at a setting of 50 for two minutes in the reinverted position. The cylindrical specimens in the stoppered plastic tubes were then placed in the fog room along with the theta specimens.

Removal of Specimens from Molds

All specimens remained in the fog room for approximately twenty hours after casting. At the end of this time the specimens were taken from the fog room and the molds removed. Each cylinder mold was immersed in a hot water bath (160 to 180^{o}F) for thirty seconds. The plastic test tube mold expanded slightly with the increase in temperature allowing the cylindrical paste specimen to slide from the mold when the mold was tapped gently on a solid surface. Teflon cylinder molds required approximately three minutes in the water bath due to mold thickness of one quarter inch. It was hoped that the brief immersion of the stoppered molds in the water bath did not exert significant thermal stresses in the paste specimens.

As each specimen was removed from its mold it was placed immediately in a saturated calcium hydroxide solution for curing. Once all specimens were removed, approximately one-half inch was cut from each end of the cylinders. The purpose was twofold. The test tube molds naturally produced a specimen with one end rounded and to have a cylindrical specimen with planar ends it was necessary to remove the rounded portion. The upper end required removal to provide a plane surface and to remove any laitance which may have been formed during the initial curing. After cutting the ends with a diamond saw the specimens were returned to the curing solution where they remained until they were tested.

The theta molds required careful removal due to the very narrow web (0.10 inches) and the relatively low strength of the paste at twenty hours. The twelve screws which fastened the two halves of the teflon mold were removed and the mold placed in the hot water bath such that the water level reached the midpoint of the mold (see Figure 2). The high coefficient of thermal expansion of the teflon made it possible for the bottom half of the mold to expand in relation to the top half and the mold separate. The top half of the mold containing the specimen was then held vertically and the perifery immersed in the hot water bath.

The teflon expanded and the specimen was easily extracted with only gentle pressure. The theta specimen, with the center inserts still in place, was then immersed in the lime water curing solution for several minutes to make sure that the specimen did not start to dry thereby creating shrinkage stresses in the specimen. To remove the center insert portions of the teflon mold, the specimen was placed in a freezer with the teflon portions resting on steel blanks. The specimen remained in the freezer at $0^{\circ}F$ for one minute and thirty seconds. After this time the inserts were removed by gentle tapping them out with a small wooden mallet. Extreme care was taken at all times to prevent straining the cement paste specimen.

Once the theta specimens were removed from the molds they were placed in lime water for curing. Each specimen was carefully examined visually to check for surface defects. The defects generally fell into two classes, one being entrapped air voids along the edges and the other being chipping which had occurred during the demolding procedures. Each specimen was taken from the lime water and the bottom and top surfaces were polished on a glass plate using No. 1200 grit. This procedure eliminated any large surface defects and any laittance formed on the exposed surface during initial curing.

Curing

The specimens from each batch were kept together and allowed to cure in lime water until immediately prior to testing.

Testing

Three theta specimens and six cylindrical specimens were made from each mix. Three of the cylinders were tested in compression and three were tested using the diametral-compression test. In order to correlate the results from the various types of tests the specimens from each mix were tested at the same age. Compression tests were conducted using a Riehle screw-gear machine with a fifty thousand pound capacity. The load at failure was determined by the position of the poise weight on the scale beam. Theta tests and diametral-compression tests were performed using a hydraulic testing machine. The testing machine is a three component unit manufactured by the MTS Division of Research, Inc. and consisting of a 20 to 40 kip load frame (Model 308.01), a load control unit (Model 483.01), and a power unit (Model 502.03). A 500 pound load cell was used when theta specimens were tested since the total load at failure was less than 200 pounds. A 5000 pound load cell was used for the diametral-compression tests. These load cells were checked with a proving ring prior to each series of tests.

Loads and vertical strains were recorded with a brushpen recorder attached to the load cell and the hydraulic system. In addition an X-Y recording unit was used to supplement the brush pen recorder. Direct plots of load versus vertical deflection were thus available for tests made on the hydraulic testing machine. The specimens were loaded using a controlled rate of vertical head travel which in the initial portion of the investigation was selected an 0.05 inch per minute, the standard concrete testing rate.

Compression Testing

The cylindrical specimens to be tested in compression were removed from the curing water and the ends polished with No. 1200 grit to provide a smooth plane surface in order to eliminate capping the specimen.

Capping could affect the strength results since the specimens were small and of relatively high strength themselves. The specimens were replaced in lime water until immediately prior to testing. The ends of the specimens were wiped to remove any excess water and the specimens positioned in the testing machine with a wet cloth surrounding the specimen to prevent drying during the test. See Figure 3 for the testing setup. The specimens were loaded under a constant rate of vertical head travel of 0.05 inches per minute and the load at failure recorded.

Diametral-Compression Test

As in the compression test the ends of the cylinders were polished to provide smooth plane surfaces. The polishing of these specimens was done at the same time as the theta specimens, at the age of twenty-four hours.

For testing the specimens were placed in a special specimen holding jig (see Figure 4) and cardboard bearing strips positioned the bearing blocks and the specimen. The specimen and jig were then placed in the testing machine and a small seating load of approximately fifty pounds was applied to the specimen. With the specimen seated the holding jig was removed and the specimen alignment checked. As in the compression test, the specimen was surrounded with a wet cloth to prevent the specimen from drying. The cloth was also effective in preventing any part of the specimen from falling from the testing machine upon failure. Load was then applied using a constant rate of head travel of 0.05 inches per minute. The load was immediately released when fracture of the specimen occurred. This precaution was taken so that the specimen would not be shattered and the fractured faces could be examined.

Theta Test

The theta specimens were removed from the curing bath and immediately placed in the testing machine. The loading ram was moved up so that the loading head just made contact with the specimen. This type of test requires that the central web of the specimen be horizontal during loading to prevent eccentricity. This was accomplished by observing the free water on the web. Since the specimen was placed in the testing machine immediately after removing it from the curing water there was enough free water on the surface of the web to act as a level. Once the specimen was leveled and seated it was loaded with a constant rate of vertical head travel of 0.05 inches per minute.

The load was released immediately when failure of the web occurred. This was done to prevent fracture of the outer portion of the specimen and possible damage to the fractured faces. The specimen was returned to the lime water. The outer ring of the theta was cut with a diamond saw which allowed the central web to be separated at the point of fracture without damage to the fractured faces. The thickness and depth of the web was then checked with a micrometer and the results recorded. The web was broken on each side of the fracture and the pieces marked to indicate the original fracture surface. These pieces were then placed in a small plastic vial for storage prior to microscopic examination of the fracture surfaces.

RESULTS

Over 150 theta specimens, 150 diametral-compression specimens, and 50 compression specimens were tested during the first portion of the investigation. A number of these specimens were manufactured while mixing, molding, demolding, and testing procedures were being developed and perfected. As would be expected, the test results for these specimens were highly variable. However, as the procedures and techniques were perfected and the operator became more proficient the variability decreased. While an analysis of a large portion of the data is not valid due to the high variability, several important factors were found to exist.

The results reported here are for mixes produced by the vacuum mixing procedures. The two main variables were the water-cement ratio and the specimen size. It has been established in other investigations that these factors are the primary ones influencing the "strength" of mortars and concretes.

Compression Test Results

Compression tests were performed on paste cylinders with a height to diameter ratio of approximately two to one. There was a slight deviation from the height due to the sawing and polishing of the ends of the specimens. Water-cement ratio was the only variable in this portion of the investigation. The compressive strength was determined to provide a basis of comparison for the diametral-compression test and the theta test. According to Griffith's original work (1), the tensile strength of a brittle material should be one-eighth of the compressive strength.

The results show that the higher the water-cement ratio the lower the compressive strength. This was expected since the water-cement ratio controls the porosity of the cement paste through its effect on the capillary pore structure. The higher the water-cement ratio the higher the capillary pore space and the lower the compressive strength. Table 2 presents a tabulation of the compressive strength results in summary form.

TABLE 2

Summary of the Compressive Strength Results

W/C	No. of Specimens	Strength, psi	Range, psi
0.4	9	14,000	12,000 - 16,200
0.5	15	9,200	7,640 - 10,100
0.6	6	6,900	6,490 - 7,090

The fracture cement paste specimens were of the typical conical shape exhibited by concrete when it fails in compression tests. There was more of a columnar type failure in the central portion of the cement paste specimens than is usually found in concrete. It was necessary carefully to position the compression specimen in the testing machine and make sure the specimens was aligned in the center of the loading blocks. Precautions were taken to provide smooth plane ends for the compression specimens since capping compounds were not used on the ends. Slight deviations in the surface smoothness of the ends cause local stress concentrations and premature failure of the specimen. Preliminary tests pointed up this problem and all compressive specimens were carefully sawed and polished to provide the smooth plane ends required.

Figure 5 shows the relationship of stress at failure to the water-cement ratio for the compression, theta and diametral-compression tests.

Results of Diametral-Compression Tests

Diametral-compression tests were performed on cylindrical cement paste specimens of various diameters and lengths. The diameters were 0.5, 1.0 and 1.1 inches. The length of the cylinders varied from over two inches to less than two-tenths of an inch. Theoretical analyses of the test and photoelastic analysis studies indicate that the length of the specimen should have no effect on the stress distribution within the specimen (9).

The two-dimensional stress analysis of the diametral-compression specimen yields the following stress equations: $\sigma_x = \frac{2P}{\pi dt}$ and $\sigma_y = \frac{-6P}{\pi dt}$ where P is the applied load, d is the specimen diameter and t is the specimen length (5). The maximum tensile stress in the central portion of the specimen is $\sigma = \frac{2P}{\pi dt}$ and the tensile stress is constant over approximately three quarters of the specimen diameter.

It should be pointed out that the maximum tensile stress in the diametral-compression specimen occurs in the portion of the specimen directly in line with the direction of load application. While fracture may originate at any point in the central plane the tensile stress decreases away from the plane and therefore failure is generally limited to the central plane where the stress is a maximum. In the theta specimen the central web has a uniform cross-section for approximately 1.5 inches of its length. The tensile stress is uniform along this length and hence the specimen can fail at any plane along the web.

The position of the diametral-compression specimen in the testing machine and the orientation of the bearing strips between the specimen and the bearing blocks is an important factor. A number of the initial tests were affected by misalignment of the specimen or the bearing strips. Placing the specimen and positioning the bearing strips while applying a small seating load was extremely difficult. Once the specimen was seated it was difficult to determine if the bearing strips were accurately placed. A technique of positioning the specimens in the testing machine with the bearing strips held in place by a specimen holder slotted to hold the bearing strips in the proper position (see Figure 4). Both the variability of the test and the operator error were reduced.

In order to make a comparison with the other test methods, the diametral-compression specimens were cast from mixes with water-cement ratios of 0.4, 0.5 and 0.6. Both theta specimens and compression specimens were made from the same mixes. The diametral-compression specimens were of the same general dimensions as the compression specimens in one phase of the investigation. The results of a portion of the diametral-compression tests are tabulated in Tables 3, 4, and 5.

The average tensile stress was 1110 psi for specimens with a water-cement ratio of 0.4, 860 psi for 0.5 and 680 psi for 0.6. The range was used to estimate the standard deviation of the data. Low values which were suspect were discarded in the calculations (this amounted to one or two extremely low values). Coefficients of variation were calculated from the data and found to be 12% for the specimens with a water-cement ratio of 0.4, 13% for the specimens with a water-cement ratio of 0.5 and 14% for the specimens with a water-cement ratio of 0.6. One possible cause of the relatively high coefficients could be the random distribution of small air voids or flaws in the specimens and another could be the nature of the test itself.

TABLE 3

Typical Results of Diametral Compression Tests on One Inch Diameter Cylinders at 28 Days

(W/C = 0.40)

Specimen No.	Length, in.	Failure Stress, psi
30 - 1	1.90	600
2	1.93	1250
4	1.86	855
31 - 1	1.91	1240
2	1.88	1190
50 - 4	2.14	1220
5	2.11	1115
6	1.58	690
52 - 1	2.13	1105
2	2.14	1010
3	2.16	970
4	2.25	860
5	2.13	1285
6	2.08	1270

$\bar{X} = 1040$ $n = 14$ $R = 1285 - 600 = 685$

Dropping the two low values:

$\bar{X} = 1110$ $n = 12$ $R = 1285 - 855 = 430$

Estimated standard deviation = 430 (0.307) = 135

Coefficient of variation = $\frac{135}{1110} \cdot 100 = 12\%$

TABLE 4

Typical Results of Diametral Compression Tests on One Inch Diameter Cylinders at 28 Days

(W/C = 0.50)

Specimen No.	Length, in.	Failure Stress, psi
39 - 1	2.02	930
2	2.04	755
3	2.08	795
40 - 1	2.14	885
2	2.15	890
3	2.14	945
41 - 1	1.98	830
2	1.95	890
3	2.14	895
42 - 1	2.13	810
2	2.11	880
3	2.09	840
44 - 1*	2.10	810
2*	2.10	1030
3*	2.14	760
46 - 1	2.13	755
2	1.96	650
3	2.10	730
49 - 1*	1.94	1000
2*	1.93	740
3*	1.61	910
51 - 1	1.84	810
2	1.82	1120
3	1.81	1110
54 - 1	2.06	800
2	2.06	820
3	1.96	665
4	2.00	685
5	1.98	1000
6	2.01	980

$\bar{X} = 860$ $n = 30$ $R = 1120 - 650 = 470$

Estimated standard deviation = 470 (0.245) = 115

Coefficient of Variation = $\frac{115}{860} \cdot 100 = 13\%$

* Tested at 29 days.

TABLE 5

Typical Results of Diametral Compression Tests on One Inch Diameter Cylinders at 28 Days

(W/C = 0.60)

Specimen No.	Length, in.	Failure Stress, psi
45 - 1	2.03	520
2	1.89	785
3	1.98	805
47 - 1	1.86	525
2	1.85	635
3	1.90	820
48 - 1	1.93	650
2	1.88	395
3	1.86	795
53 - 1	1.45	625
2	1.57	585
3	1.75	730

$\bar{X} = 655$ $n = 12$ $R = 820 - 395 = 425$

Dropping the one low value:

$\bar{X} = 680$ $n = 11$ $R = 820 - 520 = 300$

Estimated standard deviation = 300 (0.316) = 95

Coefficient of variation = $\frac{95}{680} \cdot 100 = 14\%$

Three general patterns of failure were noted in specimens tested in diametral-compression. The normal tensile failure mode is where fracture is initiated in the central plane and the specimen splits into two nearly identical halves. If the load is not removed immediately from the fractured specimen the two halves take the load and secondary failure occurs in each half. This type of failure is referred to as a "triple-cleft" failure. The calculation of tensile stress for a specimen exhibiting triple-cleft failure is valid since the secondary failure occurs after the initial failure (9). The third mode of failure noted in the specimens was a shear-type failure in the region immediately adjacent to the bearing strips. The most common cause of this type of failure is misalignment of the specimen or bearing strips which results in stress concentrations and compression failure stresses in the area of the bearing strips. Stress calculations for a specimen failing in this manner are not valid.

In order to investigate the effects of specimen size on the stress at failure, diametral-compression tests were performed on specimens of various sizes. The results of these tests indicated that the calculated stress at failure increased with decreased specimen size. The average failure stress for diametral-compression specimens with a diameter of one inch and a length of approximately two inches was roughly 60% of the stress determined for theta specimens of the same water-cement ratio. However as the central plane of failure in the diametral-compression specimen was reduced by decreased diameter and/or shorter lengths the calculated failure stress approached that of the theta specimens. A plot of specimen failure plane area versus stress is shown in Figure 6 illustrating the relationship of failure stress to the area stressed.

One hypothesis advanced to explain this behavior is based on statistical considerations. The basic assumption is that there exists in specimens a number of inherent flaws and as the size of the specimens is reduced the probability of a critical flaw being present and critically oriented is reduced. The result is that the smaller the specimen, the less the probability of failure at a given stress. In the case of hardened cement paste specimens tested under saturated conditions an additional hypothesis may be advanced. The specimens have a porosity due to the nature of the material and in a saturated condition the pores are filled with water. The permeability of cement paste is such that if a specimen is loaded part of the load is transferred to the water and a pore water pressure would be created. This is analogous to the pore water pressure in soils.

If the cross section is small enough even at low permeabilities, the pressure may dissipate or be decreased during loading. In the larger specimens the pressure cannot dissipate fast enough under loading and relatively large pore water pressures may exist. These conditions may be analogous to a "drained" and "undrained" test, respectively. The solid structure in the smaller specimens would carry most of the load while in the larger specimens the solid structure carried part and the pore water carries part of the load. The total stress is equal to the sum of the effective stress (see Ref. 10, p. 79) and the pore water pressure. If failure is the result of ruptured solid bonds, then the effective stress is of primary concern. While total stresses calculated for the large specimen may be less than that for a smaller specimen, the effective stresses may be equal.

Evidence that pore water pressures of considerable magnitude were present in the larger specimens was found. The fractured faces in the larger diametral-compression specimens were found to have a relatively large amount of free water on their surfaces. Since the permeability is low there must exist pressures of a magnitude to cause the water movement and hence pore water pressures in the paste specimen.

Results of Tests on Theta Specimens

The theta specimen shape was originally developed in an attempt to overcome the difficulties of other types of tensile tests. The specimen shape was evaluated by means of photoelastic pattern in the specimen. The stress equation for tensile stress in the web was determined to be: $\sigma = 13.8 \frac{P}{Dt}$, where σ is stress in the web, P is the applied load, D is the specimen diameter, and t is the specimen depth (6). In this investigation theta specimens with a three inch diameter were used. The stress equation becomes: $\sigma = \frac{4.6P}{t}$ for a diameter of three inches. The specimen depth varied slightly from specimen to specimen as a result of the polishing procedures used to remove surface flaws.

The water-cement ratio was considered as a variable in this test as in the compression and diametral-compression tests. As mentioned previously the tensile stress is uniform over approximately 1.5 in. of the central web. For this reason the "weakest link" theory is more applicable then in the diametral-compression test. Since the cross-section of the theta web is small, approximately 0.03 in.^{2}, any large flaw will result in failure at low stress. The results of the initial tests were highly variable because of the large amount of air entrapped during mixing. This variability was reduced by mixing the fresh paste under partial vacuum. The results of some typical test specimens vacuum mixed are presented in Tables 6, 7 and 8. The average tensile stresses for these specimens are higher than average tensile stresses determined from the diametral-compression tests. The averages were 1660 psi for a water-cement ratio of 0.4, 1400 psi for a water-cement ratio of 0.5 and 1180 psi for a water-cement ratio of 0.6.

Part of the variability of the results may be explained by the fact that there were still some air voids present in the specimen webs. Examination of the fractured faces showed that if an entrapped air void was in the failure zone the fracture plane always went through the middle of the void. Position of the specimen in the testing machine was important since eccentric loadings occur if the specimen is tilted in the machine, and a calculation of stress based on the equation $\sigma = \frac{4.6P}{t}$ is not valid.

The fractured web of each theta specimen was examined microscopically. The path of the fracture was in practically all cases in a plane at right angles to the central web. In cases where the fracture deviated from the flat plane surface it curved to fail through other entrapped air voids as well as the one at which it originated. During the microscopic examinations it was noted that a change in surface roughness occurred. Where fracture occurred through entrapped air voids, the fracture surface was relatively smooth in the vicinity of the flaw and increased in roughness as the distance from the flaw increased. This type of pattern has long been evident in studies of the fracture of metals.

The smooth area supposedly represents "slow" crack growth, in which, as the load was applied, the crack grew at a relatively slow rate (11). If the load were removed, the crack growth would stop. However, once the crack reached a critical size it would propagate with no increase in load, causing complete fracture of the specimen. The crack propagates with an

TABLE 6

Typical Results for Tests
on Theta Specimens at 28 ays

(W/C = 0.40)

Specimen No.	Thickness, mm	Failure Stress, psi
29 - 1*	9.29	920
2*	10.00	1170
3*	9.98	1630
30 - 1	8.69	1560
2	6.05	1700
3	5.50	2040
31 - 1	8.30	800
2	7.42	1465
3	3.82	1620
50 - 1	6.26	2090
2	6.97	1810
3	7.71	--
52 - 1	6.73	2170
2	8.39	1255
3	8.36	1410

$\bar{\bar{X}}$ = 1550 psi n = 14 R = 2170 - 800 = 1370

Dropping the two low values:

$\bar{\bar{X}}$ = 1660 n = 12 R = 2170 - 1170 = 1000

Estimated standard deviation = 1000 (0.307) = 307

Coefficient of variation = $\frac{307}{1660} \cdot 100 = 19\%$

* Tested at 29 days

TABLE 7

Typical Results for Tests on Theta Specimens at 28 Days

(W/C = 0.50)

Specimens No.	Thickness, mm	Failure Stress, psi
39 - 1	--	--
2	8.29	1200
3	8.34	1080
40 - 1	9.11	705
2	9.29	1070
3	7.80	1230
41 - 1	7.44	1790
2	8.31	1350
3	7.63	1270
42 - 1	9.31	1040
2	--	--
3	9.30	1090
43 - 1*	8.32	1825
2*	8.16	1145
3*	8.84	1610
44 - 1*	8.32	1195
2*	6.05	1755
3*	8.14	1535
46 - 1	8.01	1270
2	7.88	1480
3	7.50	1560
49 - 1*	7.96	1220
2*	7.73	1360
3*	9.05	1020
51 - 1	6.26	1810
2	6.97	1390
3	7.71	1760
54 - 1	7.23	1695
2	--	--
3	8.05	1670

$\bar{X} = 1370$ $\quad$ n = 27 $\quad$ R = 1825 - 705 = 1120

Dropping the one low value:

$\bar{X} = 1400$ $\quad$ n = 26 $\quad$ R = 1825 - 1020 = 805

Estimated standard deviation = 805 (0.256) = 206

$$\text{Coefficient of variation} = \frac{206}{1400} \cdot 100 = 15\%$$

* Tested at 29 days

TABLE 8

Typical Results for Tests
on Theta Specimens at 28 Days

(W/C = 0.60)

Specimen No.	Thickness, mm	Failure Stress, psi
45 - 1	8.34	925
2	7.57	1340
3	8.26	1400
47 - 1	6.87	1275
2	7.41	1180
3	6.80	1255
48 - 1	8.31	730
2	9.11	1065
3	7.28	1235
53 - 1	6.21	940
2	6.03	1260
3	5.67	1135

$\bar{X} = 1150$ $n = 12$ $R = 1400 - 925 = 670$

Dropping the one low value:

$\bar{X} = 1180$ $n = 11$ $R = 1400 - 925 = 475$

Estimated standard deviation = 475 (0.316) = 150

Coefficient of variation = $\frac{150}{1180} \cdot 100 = 13\%$

increasing velocity until a limiting value is reached. At this point the crack branches and branches again and again. This branching causes the rough surface (11). It is possible to estimate the critical crack size from the pattern present on the fracture face. The pattern can also indicate where the crack originated. In the theta specimens, this general type of pattern, smooth to rough, was found on fractured surfaces in all cases where fracture occurred through an entrapped air void.

The larger the entrapped air void the larger the initial size of a crack originating from the void. Therefore, according to the Griffith theory the stress to cause fracture will be less than if there were no flaw in the specimen. Figures 7 and 8 illustrate the relationship between void size and the tensile stress at failure. The diameter of the largest void present on the fractured face is plotted against the tensile stress at failure. The size of the voids was determined by microscopic examination of fractures surfaces of the theta webs. There is considerable scatter in the data, part of which may be attributed to the fact that the air void itself is not the critical crack at failure. Slow crack growth will occur until a critical size is reached. There were also other smaller voids present in some of the specimens and these contributed to failure.

Figures 9, 10 and 11 are photo-micrographs taken of the fractured surfaces from three different theta specimens. In Figure 9 an air void is shown on one edge of the web. The surface roughness increases from left to right. Figures 10 and 11 show air voids in different locations within the webs of two other specimens.

Results of Tests on Cylinders with Artificial Flaws

The examination of the fractured surfaces of cylinders tested in diametral-compression showed a characteristic pattern on the surface. Lines appeared on the surface curving away from one end of the specimens. These hackle marks, have been observed in other investigations studying fracture in metals and radiate away from the source of the fracture. They are caused by elastic waves rebounding from the exterior boundaries of the specimen as the crack propagates. Figure 12 shows these hackle marks as they appear on a specimen with no apparent defect. In the specimens where no defect was apparent the hackle originated at one end or the other, indicating failure began there. The surface showed a relatively smooth area grading into a rougher area as in the theta specimens. In cylindrical specimens where entrapped air voids occurred in the central plane the hackle marks radiated in all directions from the void.

In an effort to learn more about the fracture in the cement paste, artificial flaws were placed in several cylindrical specimens. Teflon disks and holes drilled in the ends of the specimens were used as flaws. Figure 14 shows a specimen which contained a teflon disk with a diameter of 0.9 inches. The hackle marks radiate away from the location of the disk in all directions indicating failure was initiated at the disk. Figure 13 shows a specimen with a small hole drilled a short distance into the end. Again the hackle marks radiate away from the artificial flaw indicating that failure was initiated from that point. In general the strength of the specimens with artificial flaws was slightly lower than those without obvious defects. However, only a few specimens were tested and more work is needed before definite conclusions can be made.

Comparisons of Test Results to Theory

Griffith's theory for fracture of a brittle material considers that pre-existing cracks are present in the material prior to loading. Griffith arrived at the equation $\sigma^2 = \frac{2E\gamma}{\pi c}$ relating stress at failure to the crack length. In the equation σ is the stress at failure, E is the Young's modulus, γ is the surface free energy of the material and 2c is the length of the internal crack (or c is the length of a surface crack). Sack modified the equation to the form $\sigma^2 = \frac{\pi E\gamma}{2c}$ for a penny shaped crack in a three dimensional analysis.

In hardened cement paste the surface tension of the material is not known. However, if the value of the surface tension of tobermorite is used, an estimate of the critical crack length can be obtained. Assuming $E = 3 \times 10^6$ psi, $\gamma = 400$ ergs/cm^2 and assuming the crack length 2c to be equal to 0.02 inches an estimate of the stress at failure may be obtained using Sack's equation.

$$\sigma^2 = \frac{\pi E\gamma}{2c} = \frac{3.14 \times 3 \times 10^{-6} \text{ psi} \times 400 \text{ ergs/cm}^2}{2 \times 0.01 \text{ inches}}$$

Since: 1 erg = 2.248×10^{-6} pound-cm and 1 in = 2.54 cm

$$\text{Then: } \sigma^2 = \frac{9.42 \times 10^6 \text{ psi} \times 400 \text{ ergs/cm}^2 \times 2.248 \times 10^{-6} \text{ lbs-cm} \times 2.54 \frac{\text{cm}}{\text{in}}}{2 \times 0.01 \text{ inch}}$$

And $\sigma^2 = 1{,}080{,}000$ or $\sigma = 1040$ psi

For a cement paste specimen with a water-cement ratio of 0.4 the stress value at failure, for a specimen with an air void size of 0.02 inches, is approximately 1400 psi. This compares to the calculated value of 1040 psi when Sack's modification of Griffith's equation is is used ($\sigma^2 = \frac{\pi E \gamma}{2c}$). A portion of the error can be attributed to estimates of E and γ .

Calculations of stress considering capillary pores as the critical crack give values much higher than those observed. If, however, entrapped air voids are considered as critical cracks the calculated stress approaches the observed stress. From observations made during the initial portion of the investigation it appears plausible that air voids are present in the cement paste in sizes ranging from those observable to the naked eye to sizes approaching capillary pore size. The relative amount may be extremely small so as to have little effect on porosity but still have an effect on the tensile strength of the specimen. It takes only one flaw, properly oriented, to initiate failure.

SUMMARY

The results of this investigation to date may be summarized as follows:

1. Mixing, molding, demolding, testing techniques and procedures have been developed for cement paste specimens used to study crack origin, growth, and inhibition.
2. A correlation exists between flaw size and tensile stress at failure.
3. Excess pore water pressure appears to be present in specimens tested in diametral-compression.
4. Fracture patterns on the fractured surfaces of both theta and diametral-compression specimens indicates the crack origin and direction of propagation.
5. If air voids ranging from those observable to the naked eye down to capillary pore size are assumed to be present in the cement paste then these may be the source of critical cracks. Calculation of stress using Griffith's equation and assuming air voids as critical cracks give values which are of the same order of magnitude as the stresses observed in tests.

REFERENCES

1. Griffith, A. A., "The Phenomena of Rupture and Flow in Solids," Phil. Trans. Royal Society of London, Vol. 221, 1920, pp. 163-198.

2. Orowan, E., "Energy Criteria of Fracture," Welding Research Supplement, March 1955, pp. 157-S-160-S.

3. Kaplan, M. F., "Crack Propagation and the Fracture of Concrete," Journal of the ACI, Vol. 58, No. 5, pp. 591-610, November 1961.

4. Irwin, George R., "Fracturing and Fracture Mechanics," T. and A. M. Report No. 202, Department of Theoretical and Applied Mechanics, University of Illinois, October 1961.

5. Mitchell, Neal B., Jr., "The Indirect Tension Test for Concrete," Materials Research and Standards, Vol. 1, No. 10, pp. 780-788, October 1961.

6. Durelli, A. J., Morse, S., and Parks, V., "The Theta Specimen for Determining Tensile Strength of Brittle Materials, "Materials Research and Standards, ASTM, Vol. 2, No. 2, pp. 114-117, February 1962.

7. Antrim, J. D., "A Study of the Mechanism of Fatigue in Cement Paste and Plain Concrete," Ph.D. Thesis, Purdue University, January 1964.

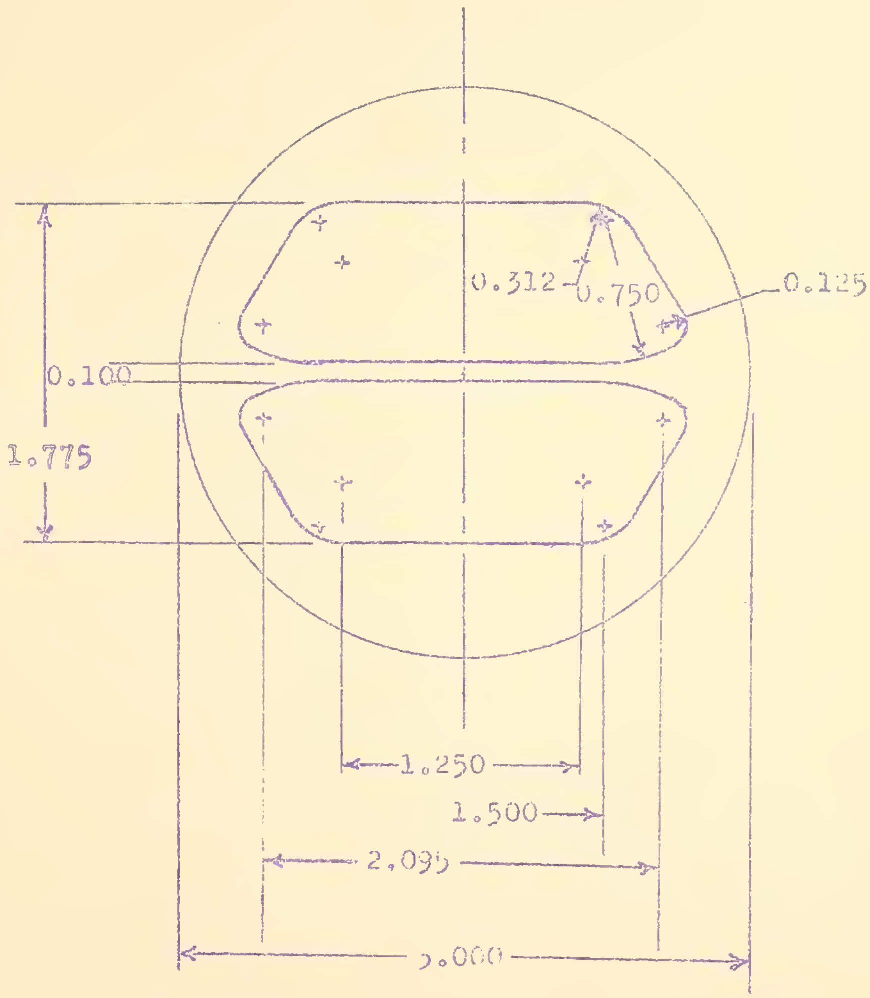
8. Mullen, W. G., "Creep of Portland Cement Paste," Ph.D. Thesis, Purdue University, June 1963.

9. Rudnick, A., Hunter, A. R. and Holden, F. C., "An Analysis of the Diametral-Compression Test," Materials Research and Standards, ASTM, Vol. 3, No. 4, pp. 283-289, April 1963.

10. Wu, T. H., "Soil Mechanics," Allyn and Bacon, Inc., 1966.

11. Corten, H. T. and Park, F. R., "Fracture," International Science and Technology, 15: 24-36, 1963.

APPENDIX



Scale: 1 inch = 1 inch

FIGURE 1. THETA SPECIMEN GEOMETRY

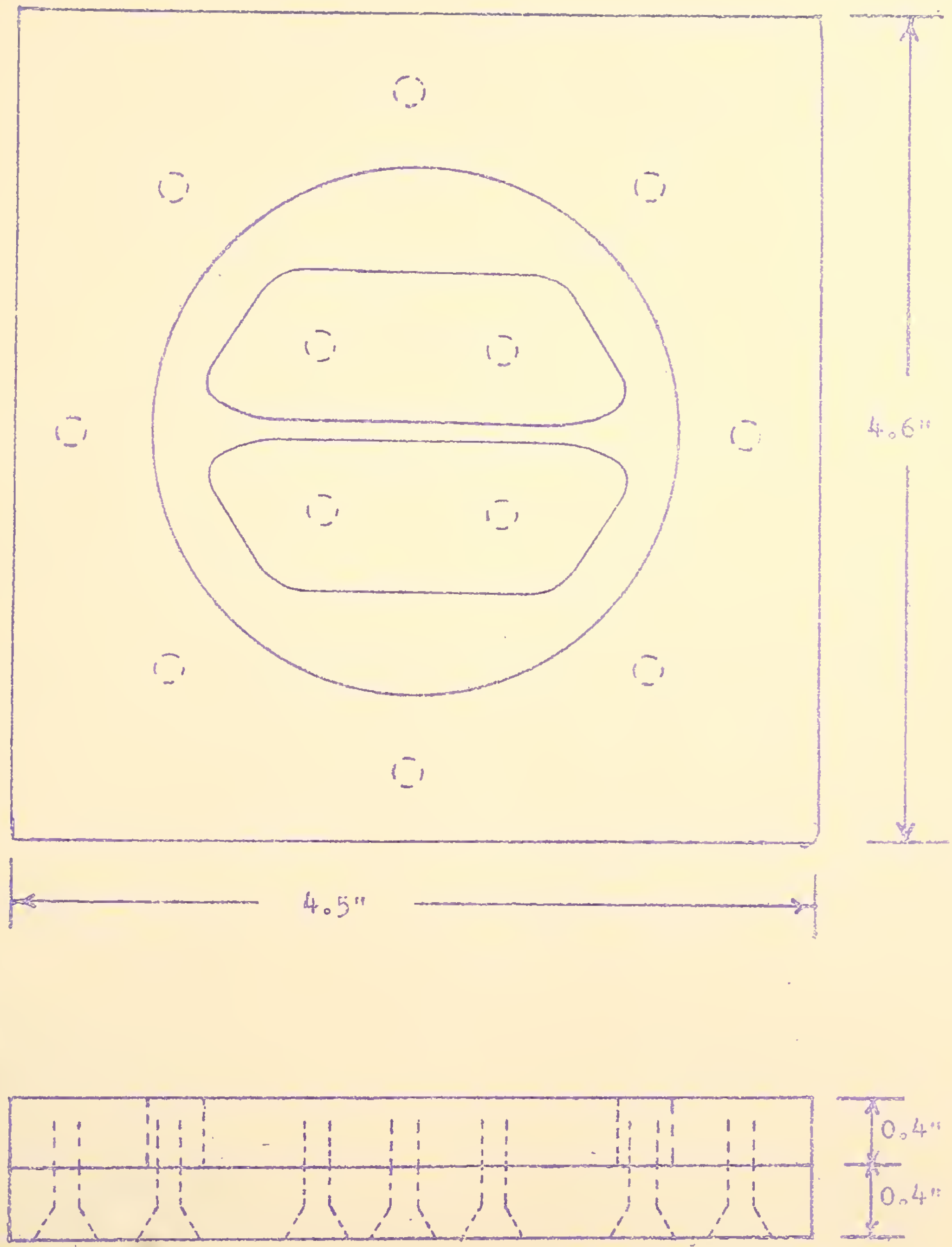


FIGURE 2: TEFLON THETA SPECIMEN MOLD

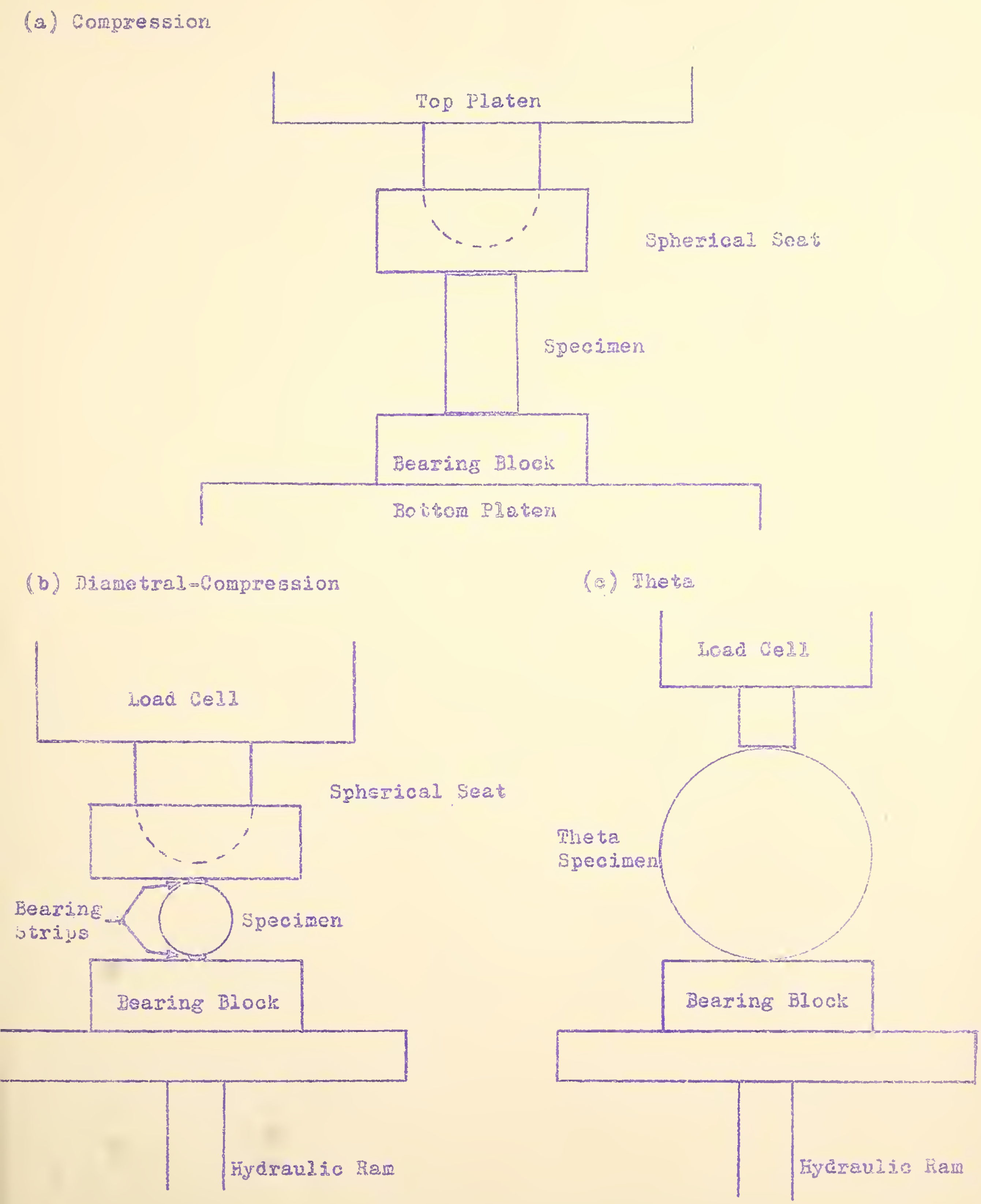


FIGURE 3. SPECIMEN TESTING ARRANGEMENTS

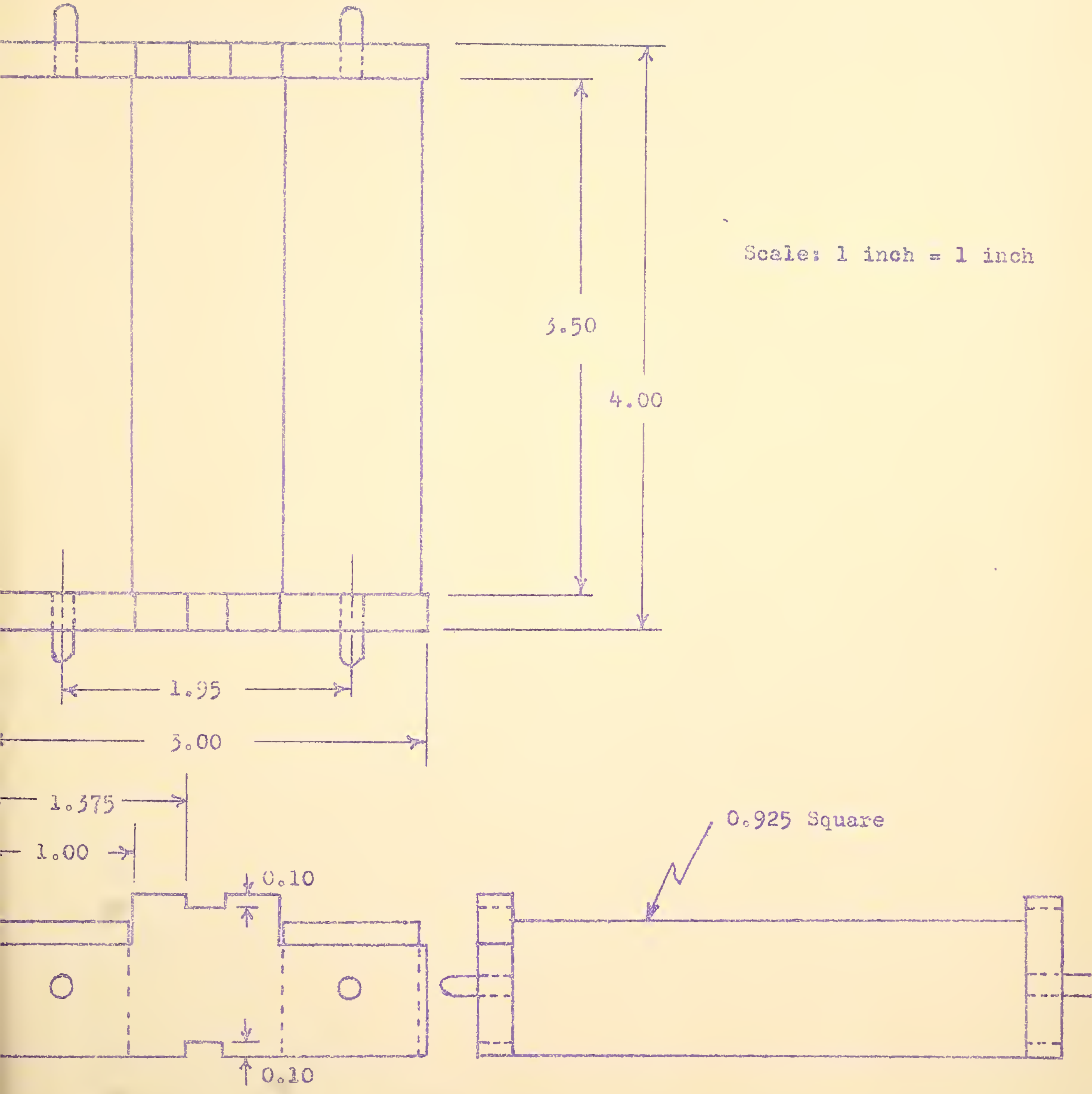


FIGURE 4. DIAMETRAL-COMPRESSION SPECIMEN HOLDER

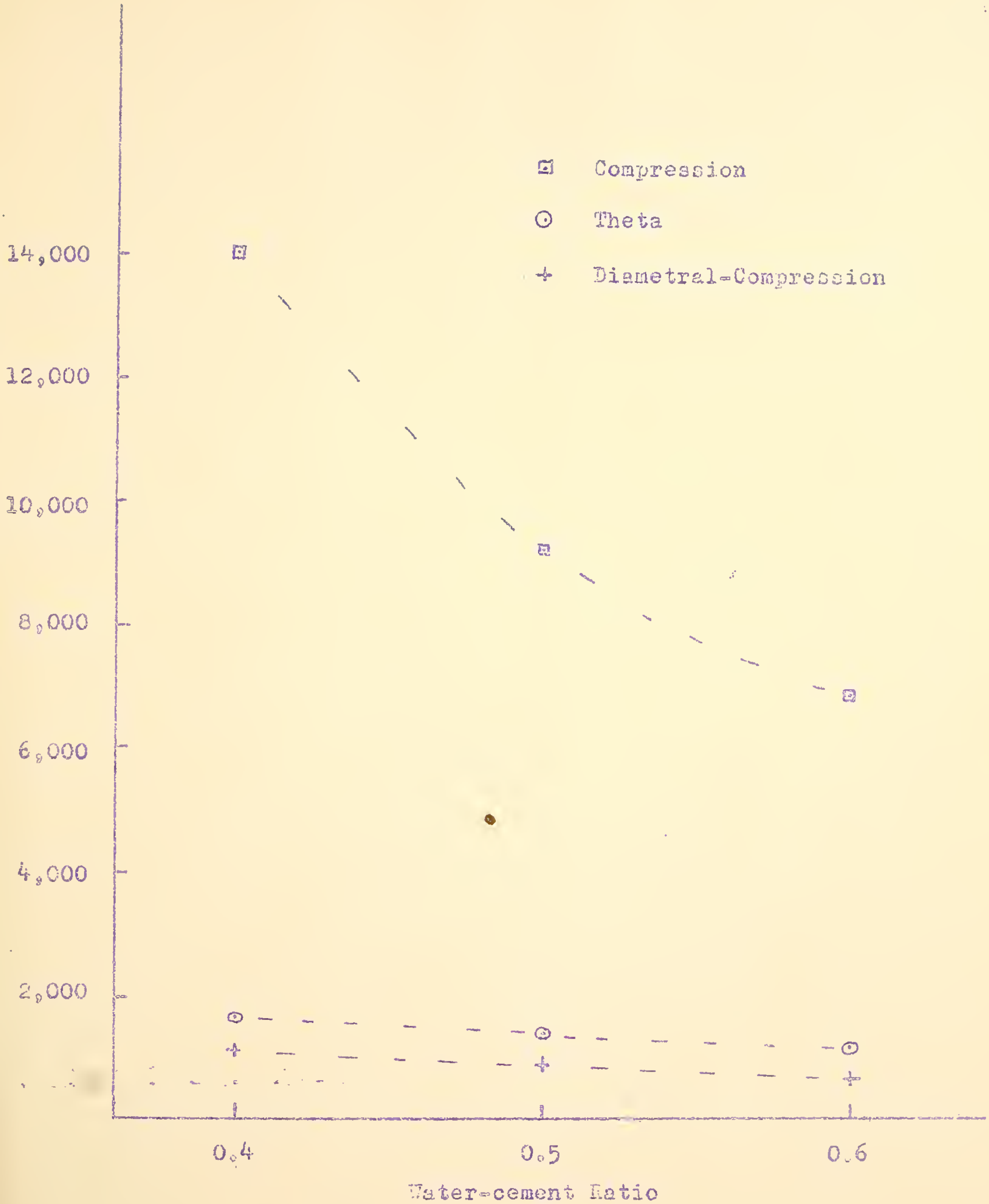


FIGURE 5. STRESS VERSUS WATER-CEMENT RATIO

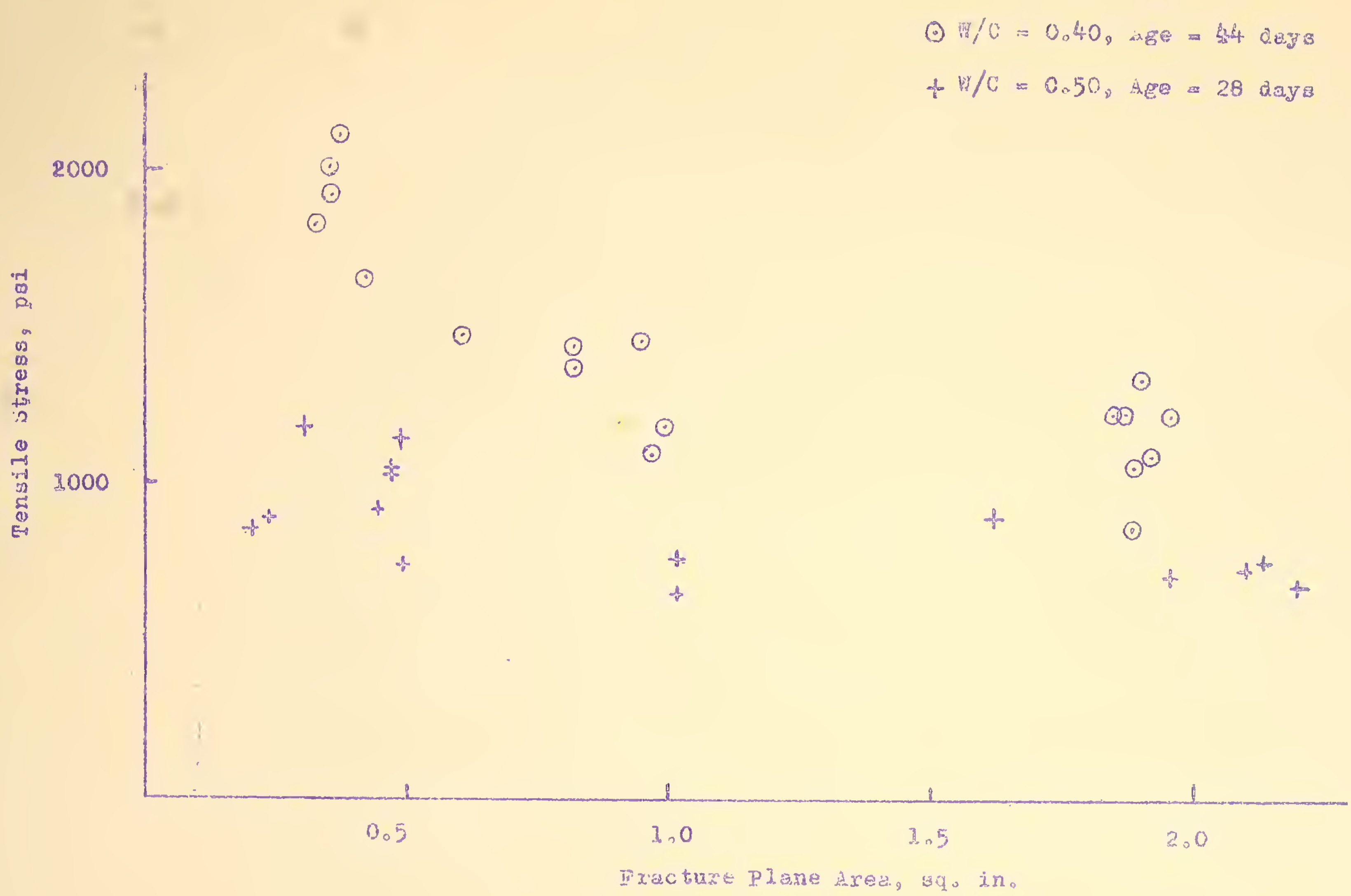


FIGURE 6. DIAMETRAL-COMPRESSION TENSILE STRESS VERSUS FRACTURE PLANE AREA

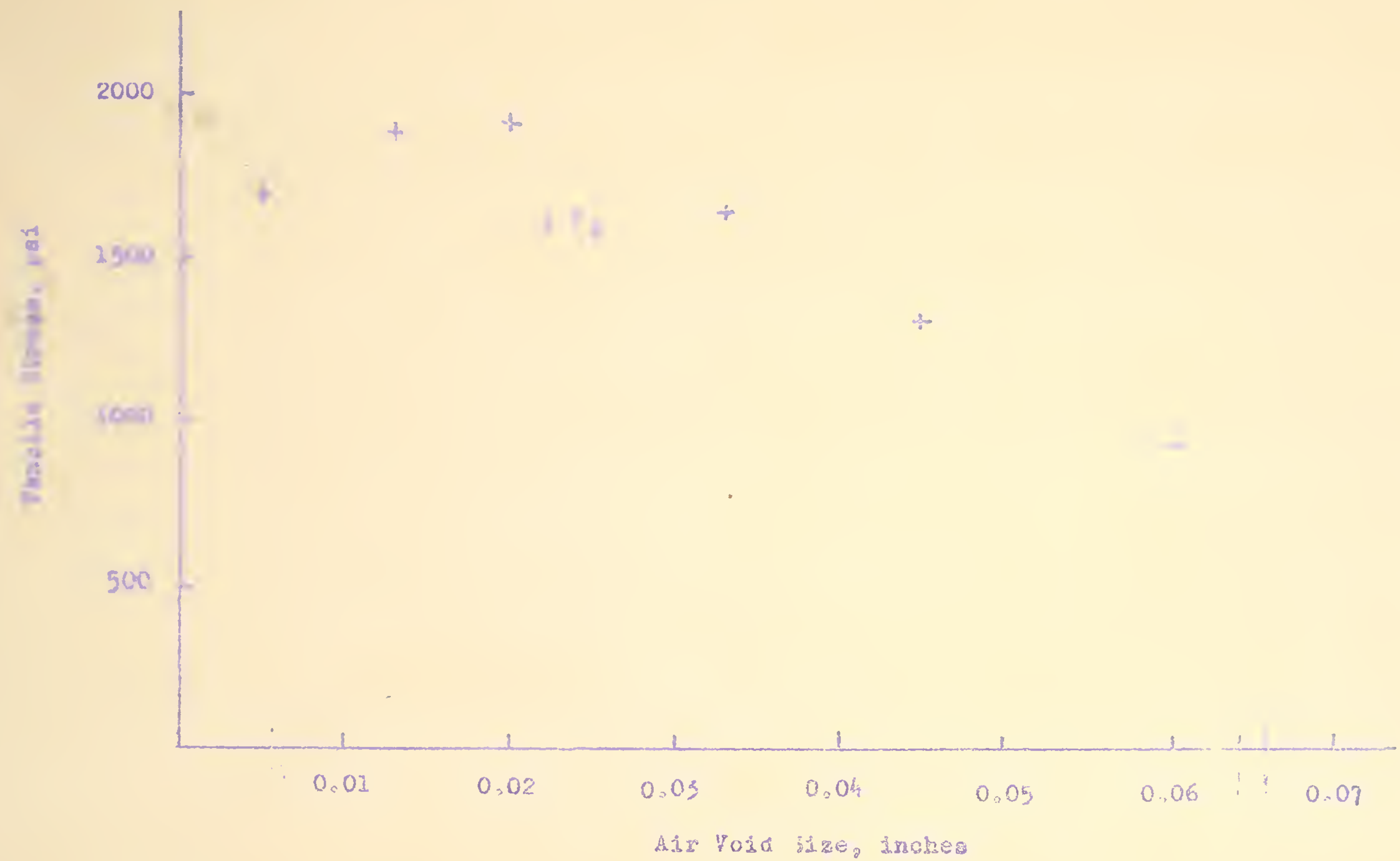


FIGURE 7. RELATIONSHIP OF AIR VOID SIZE TO STRESS, W/C = 0.3

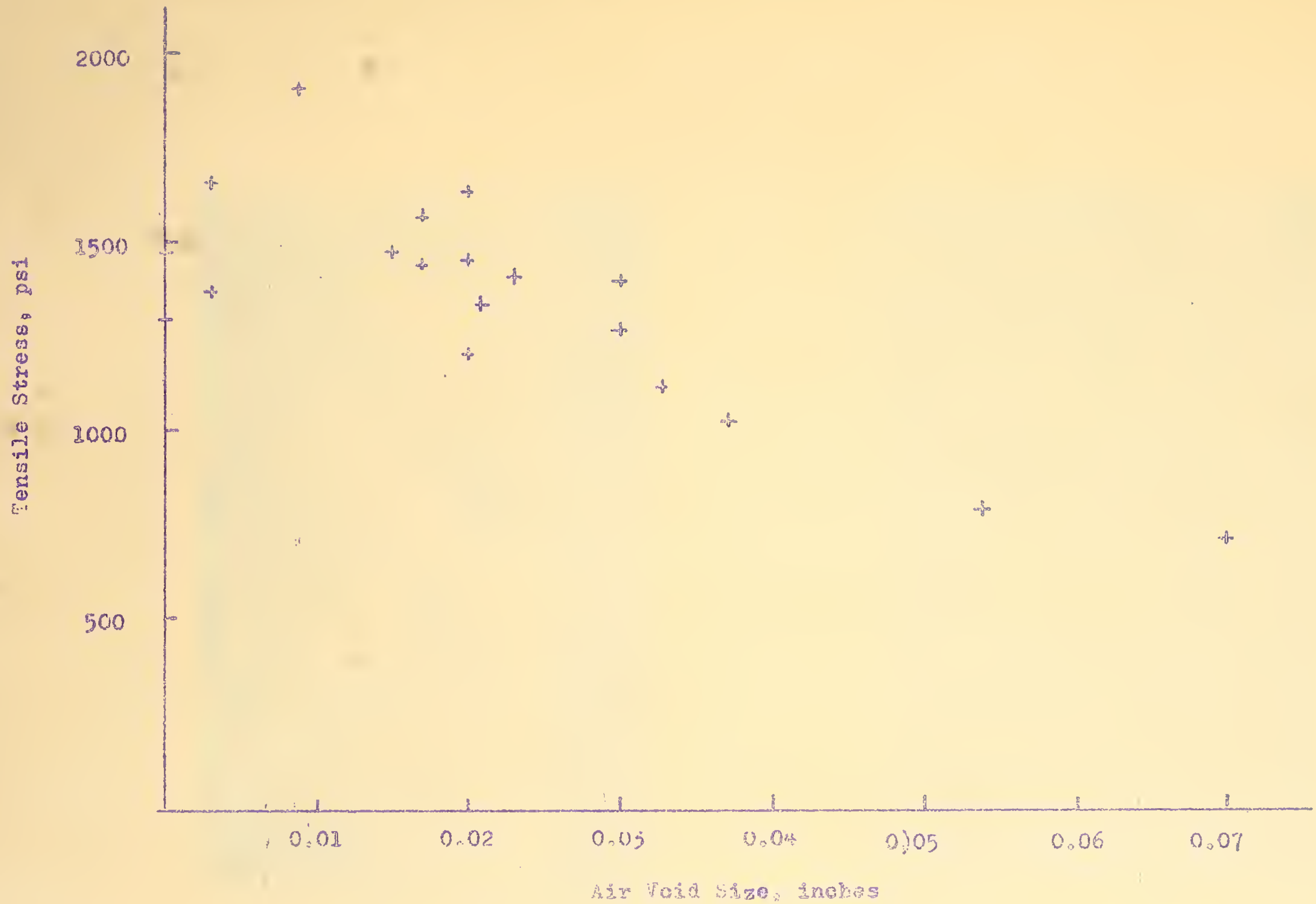


FIGURE 8. RELATIONSHIP OF AIR VOID SIZE TO STRESS w/c = 0.4

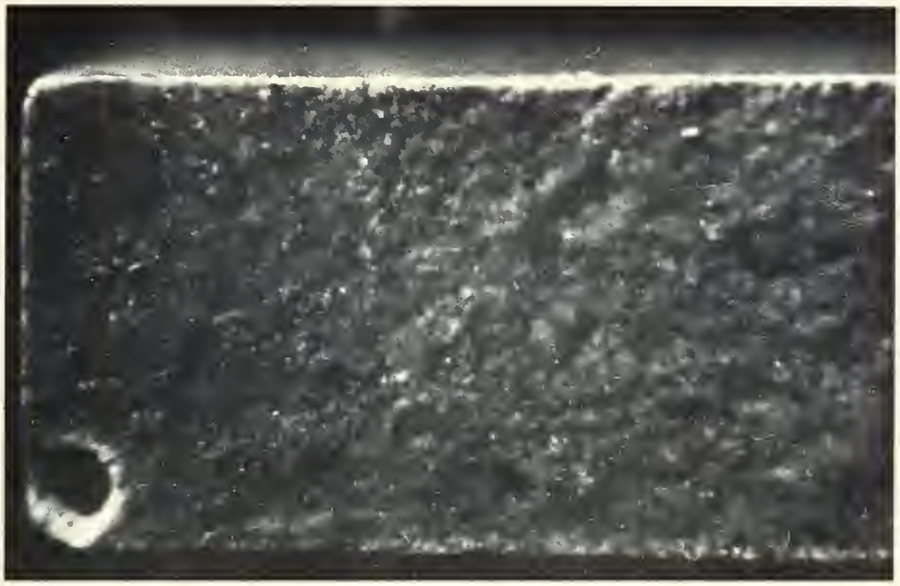


FIGURE 9. THETA SPECIMEN NO. 17-5 (x 22)

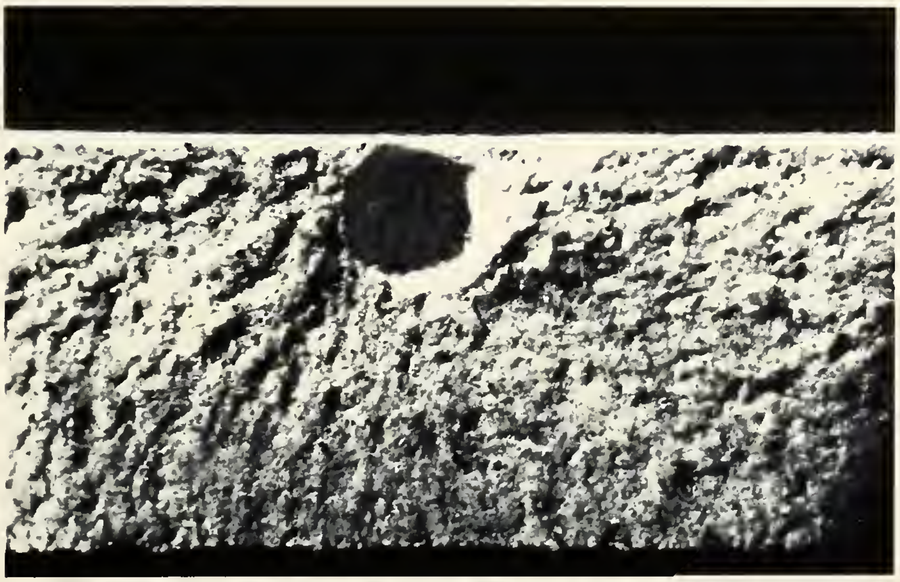


FIGURE 10. THETA SPECIMEN NO. 29-2 (x 22)

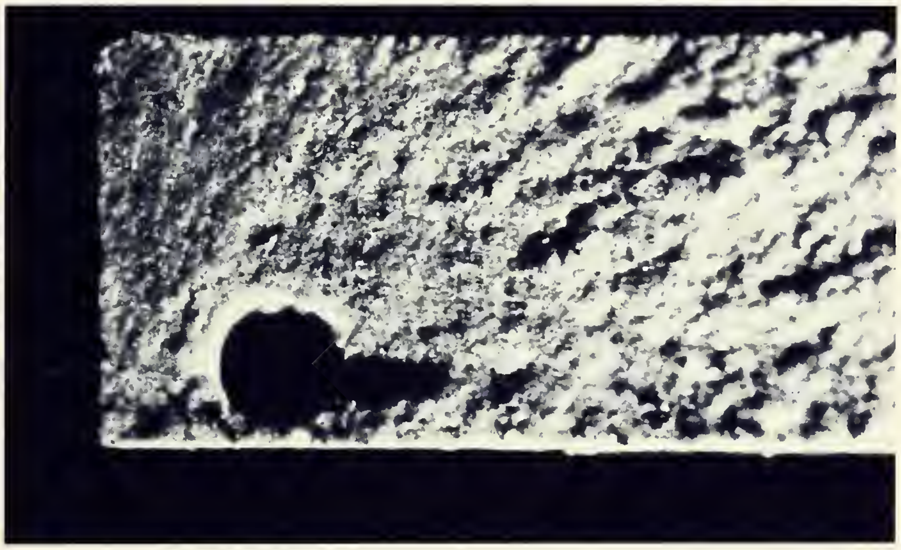


FIGURE 11. THETA SPECIMEN NO. 31-2 (x 22)

FIGURE 12. DIAMETRAL-COMPRESSION SPECIMEN NO. 46-1

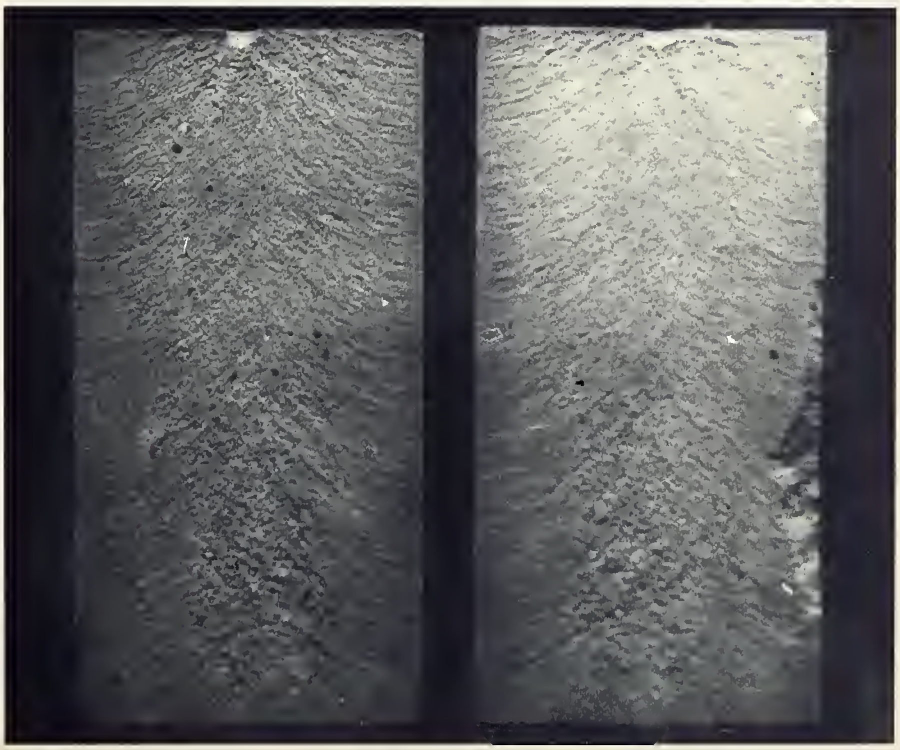


FIGURE 13. DIAMETRAL-COMPRESSION SPECIMEN NO. 4[illegible]-[illegible]

FIGURE 14. DIAMETRAL-COMPRESSION SPECIMEN NO. 50-3